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C. S. Ray

Missouri University of Science and Technology, csray@mst.edu

Sabhayu Sen

Signo Tadeu Dos Reis

Missouri University of Science and Technology, reis@mst.edu

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Characterization and Glass Formation of JSC-1 Lunar and Martian Soil Simulants

Chandra S. Ray¹, Signo T. Reis¹, Subhayu Sen²

¹*Graduate Center for Materials Research, University of Missouri-Rolla, Rolla, MO 65409*

²*BAE Systems, Marshall Space Flight Center, National Aeronautics and Space Administration, Huntsville, AL 35812*

Abstract. The space exploration mission of NASA requires human and robotic presence for long duration beyond the low earth orbit (LEO), especially on Moon and Mars. Developing a human habitat or colony on these planets would require a diverse range of materials, whose applications would range from structural foundations, (human) life support, (electric) power generation to components for scientific instrumentations. A reasonable and cost-effective approach for fabricating the materials needed for establishing a self-sufficient human outpost would be to primarily use local (in situ) resources on these planets. Since ancient times, glass and ceramics have been playing a vital role on human civilization. A long term project on studying the feasibility of developing glass and ceramic materials has been undertaken using Lunar and Martian soil simulants (JSC-1) as developed by Johnson Space Center. The first step in this on-going project requires developing a data base on results that fully characterize the simulants to be used for further investigations. The present paper reports characterization data of both JSC-1 Lunar and JSC Mars-1 simulants obtained up to this time via x-ray diffraction analysis, scanning electron microscopy, thermal analysis (DTA, TGA) and chemical analysis. The critical cooling rate for glass formation for the melts of the simulants was also measured in order to quantitatively assess the glass forming tendency of these melts. The importance of the glasses and ceramics developed using in-situ resources for constructing human habitats on Moon or Mars is discussed.

Keywords: Lunar and Martian soils simulants, Glass formation, XRD, DTA and Mössbauer.

PACS: 61.05.cp, 61.05.or, 61.43.Fs, 64.70.dg, and, 65.40.-b.

INTRODUCTION

Establishing a permanent, self sufficient and safe human habitat on planetary bodies beyond the low earth orbit (LEO), especially Moon and Mars, is critical to the success of NASA's space exploration mission. Some of the essential items for developing an extra-terrestrial habitat would require a diverse range of materials, whose applications would range from structural foundations, (human) life support, (electric) power generation to components for scientific instrumentations. In-situ processing and utilization of resources available on these non-terrestrial planetary bodies would be a reasonable and cost effective approach for developing materials needed for this effort. This approach would reduce significantly the level of up-mass and, hence, reduce the overall work load and cost of the exploration mission.

A long term effort aimed primarily at developing glass and ceramic materials using JSC-1 Lunar and Martian soil simulants, and exploring their various application potentials on the respective planetary surface has been undertaken (Ray et al., 2006; Sen, Ray, and Reddy, 2005). The importance of glasses and ceramics on the progress of human civilization is well known. Examples of their applications range from common structural components (building and sealing, composites, containers, automotive components, substrates for solar cell and electronic applications, rocket nozzles, heat resistant ceramic tiles for space vehicles) to such sophisticated applications as laser and photonic devices, optical glass fibers, gas sensors, bio-materials, electronic ceramics, and many more. The premise of this ongoing research program is to investigate what type of glass and ceramic materials can be developed from the Lunar and Martian soil compositions, and to explore the potential application areas of these materials through

extensive property analysis and characterization. The starting raw materials for developing these special glass and ceramic materials are the JSC-1 Lunar and JSC Mars-1 simulants developed by Johnson Space Center, NASA at Houston, TX.

The primary objectives of the current work are to understand the nature and type of the starting raw materials (the simulants in this case) using standard characterization procedures, and to develop a data base on the characterization results of the simulants. This knowledge is essential if one wants to develop materials having specific properties of practical interest. This paper reports the results obtained up to this time for both JSC-1 Lunar and JSC Mars-1 simulants as characterized by x-ray diffraction analysis (XRD), scanning electron microscopy (SEM), differential thermal and thermo-gravimetric analysis (DTA/TGA), chemical analysis for composition by inductively coupled plasma atomic absorption spectroscopy (ICP-AES), and Mössbauer spectroscopy. The critical cooling rate for glass formation (R_c) that determines the ability of a melt to form glass has been measured and reported for both melts.

RESULTS

The results obtained for the Lunar and Martian soil-simulants using various characterization techniques are described and discussed in the following sections.

Chemical Analysis (ICP-AES)

The compositions for the as-received JSC-1 Lunar and JSC Mars-1 simulants analyzed by ICP-AES are given in Table 1 along with the compositions reported (McKay et al., 1994; Allen et al., 1998) in the literature for these simulants. No separate determination was made for FeO in the ICP-AES analysis, and the total iron oxide content is shown as Fe₂O₃. This is indicated by the arrows in Table 1. The Lunar soil simulant, JSC-1 Lunar, was developed from a volcanic ash deposit located in the San Francisco volcano field near Flagstaff, Arizona. This ash, which was erupted from the vents in the south flank of Merriam Crater was mined, processed, and stored for future use. This JSC-1 Lunar simulant is reported to closely resemble to the low-TiO₂ Lunar soil collected from the Maria geological terrain of the Moon by the Apollo 14 Mission (Vaniman et al., 1993; Duke and Eckart, 1999). The source for the JSC Mars-1 simulant is from a volcanic ash in Hawaii, which is reported (Allen et al., 1998) to resemble the Martian regolith at the Pathfinder landing site. The results in Table 1 show that the compositions determined by ICP-AES for both simulants in the present work are very close to those reported in the literature.

TABLE 1. Average wt% Composition of the Major Constituents in JSC-1 Lunar and JSC Mars-1 Simulants.

Major Constituent Oxides	JSC-1 Lunar		JSC Mars-1	
	McKay et al., 1994	ICP-AES Present work	Allen et al., 1998	ICP-AES Present work
SiO ₂	47.7	46.0	43.5	41.7
Al ₂ O ₃	15.0	17.3	23.3	24.4
CaO	10.4	10.3	6.2	6.6
MgO	9.0	7.1	3.4	3.2
Fe ₂ O ₃	3.4	11.8	11.8	16.2
FeO	7.4		3.5	-
Na ₂ O	2.7	3.4	2.4	1.5
TiO ₂	1.6	1.9	3.8	4.0

XRD and SEM

The major components of both Lunar and Martian regoliths consist of the fragments of rocks, minerals, and glasses, although the relative proportion of the components varies widely with the site or location. The major group of minerals found (Duke et al., 1999) on the Lunar surface consists of silicate minerals, namely, olivine [(Mg, Fe)₂SiO₄], pyroxene [(Ca, Mg, Fe) (Si, Al)₂O₆], and plagioclase [(Na, Ca) (Si, Al)₄O₈], and non-silicate minerals, such as ilmenite [FeTiO₃]. In developing a simulant, the attention is paid to ensure that the relative proportion and the average size distribution of the constituent components are close to those of the regolith at a specific site of the respective planet. For example, the JSC-1 Lunar and JSC Mars-1 simulants have been developed to resemble the

regoliths collected from Maria terrain on Moon by the Apollo 14 Mission and from Pathfinder landing site on Mars, respectively.

The XRD patterns for the as-received Lunar and Martian simulants are shown in Figs. 1 (a) and (b), respectively. Major crystalline phases present in the simulants have been identified and marked in the figures. The presence of olivine, pyroxene, and ilmenite is clearly observed in the XRD of Lunar simulant. The XRD of the Martian simulant shows the presence of pyroxene and an ortho-pyroxene [(Mg, Fe) Si₂O₆] phase. Fig. 2 shows the SEM of the Lunar simulant after imbedding and polishing the (simulant) powder in a polymer matrix. All the minerals, olivine, plagioclase and pyroxene, and a glass phase have been clearly identified.

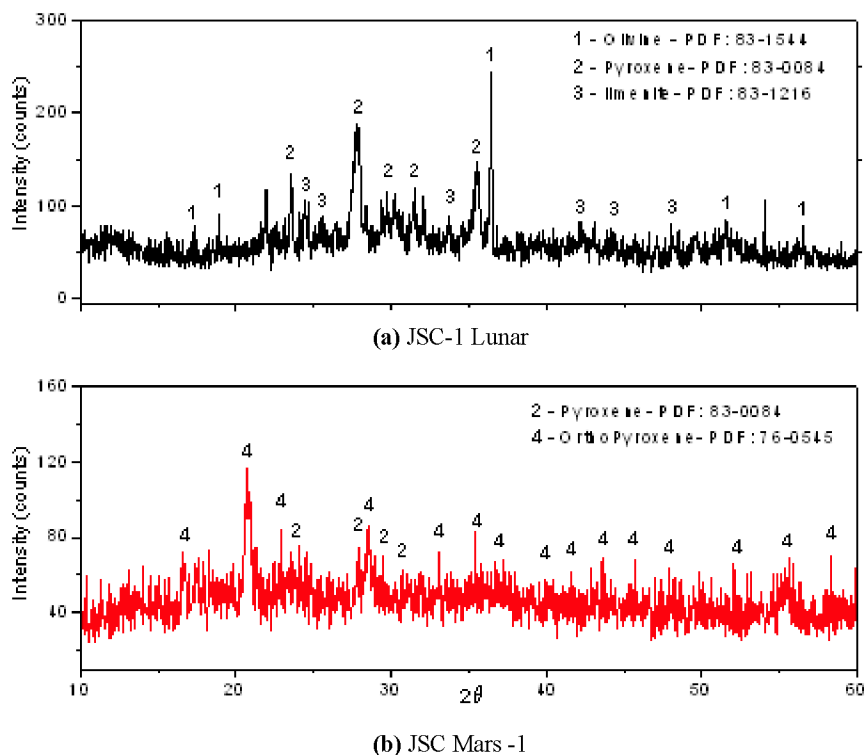


FIGURE 1. XRD Spectra for the As-Received Simulants.

DTA and TGA

A Netzsch, STA 409, apparatus was used to perform DTA experiments for the as-received Lunar and Martian simulants. The experiments were performed at a heating rate of 10 °C/min in flowing dry air from room temperature to 1300 °C for the Lunar simulant and to 1500 °C for the Martian simulant. The thermo-gravimetric (TGA) experiments were performed in a Netzsch TGA apparatus (STA 409) at a heating rate of 5 °C/min in flowing dry air up to a temperature of 1300 °C. The DTA and TGA thermal profiles for the Lunar and Martian simulants are shown in Figs. 3 and 4. The DTA profiles of both simulants, Figs. 3(a) and 4(a), show the presence of a small endothermic peak, at about 684 °C for the Lunar simulant and 925 °C for the Martian simulant, followed by an exothermic peak, at about 900 °C for the Lunar simulant and 1200 °C for the Martian simulant. The endo- and exothermic peaks correspond to the glass transition (T_g) and glass crystallization (T_c) events, respectively, which confirm the presence of a small amount of glass phase in both simulants. The endothermic peak (T_m) occurring after the crystallization event, at about 1140 °C for the Lunar simulant and at a temperature >1350 °C for the Martian simulant, corresponds to melting of the simulant. It should be noted that a complete melting peak (endothermic) was observed for the Lunar simulant (Fig. 3a), where as, it could not be achieved for the Martian simulant (Fig. 4a) even at a temperature of 1500 °C. Thus, it appears that the Martian simulant is more difficult to melt than the Lunar simulant.

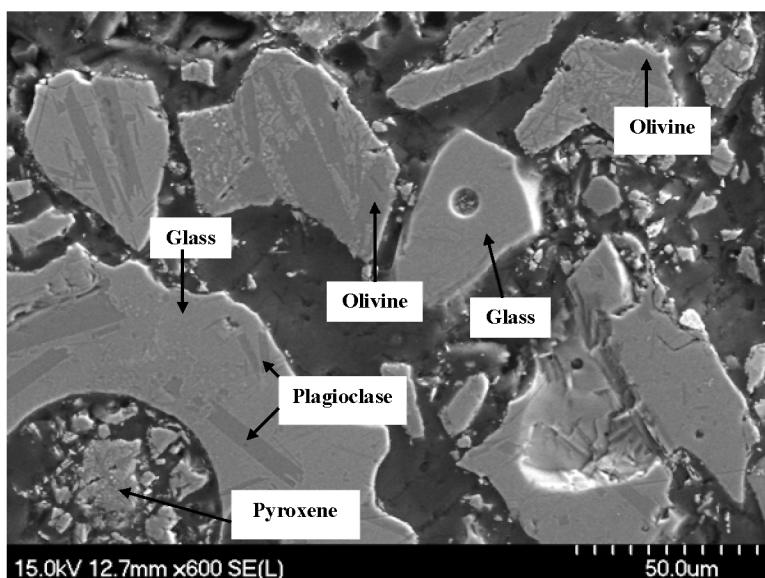


FIGURE 2. SEM Back Scattered Electron Image of JSC-1 Lunar Simulant.

The TGA profile in figures 3(b) and 4(b) is nearly featureless for both samples throughout the entire temperature range of experiment, being almost parallel to the temperature axis. This indicates that the samples neither gain nor lose any mass with temperature change, which leads to suggest that neither sample contains any volatiles or undergoes any phase transformation that is associated with a change of mass with increasing temperature.

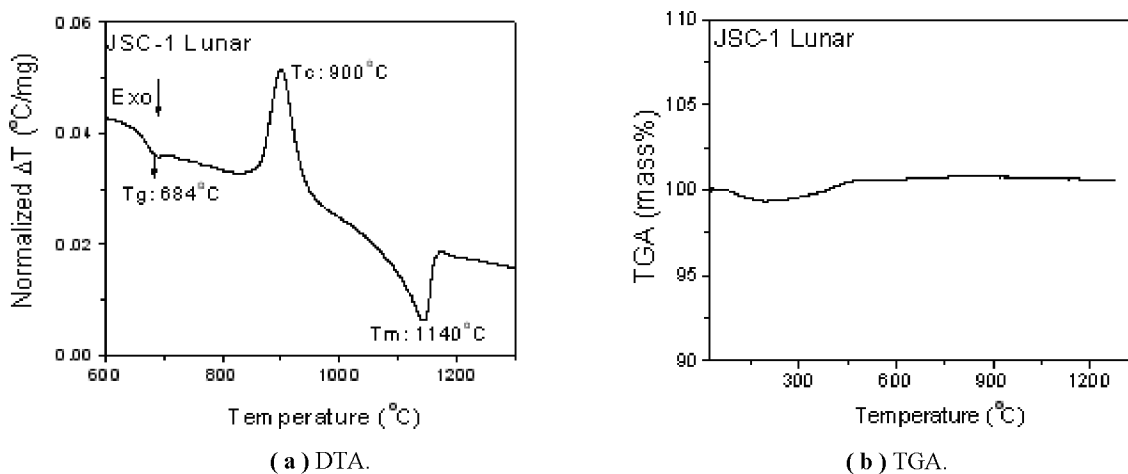


FIGURE 3. Thermal Profiles for the JSC-1 Lunar S

Mössbauer Analysis

The Lunar and Martian soils both contain a substantial amount of multivalent iron, and can be present in 0 (elemental), 2⁺, and 3⁺ oxidation states. The relative proportion of iron with different oxidation states in the planet's soil depends upon the particular atmospheric condition of the planet. For example, the Lunar atmosphere is described as hard vacuum (McKay et al., 1994) where the gas concentration varies from 10⁻⁵ to 10⁻⁴ molecules/cm³. This is approximately 14 orders of magnitude less than in the Earth's atmosphere. In the absence of any atmosphere, and hence oxygen, the actual Lunar regolith from any site explored up to this time was not found to contain iron in its 3+ oxidation state, i.e. as Fe₂O₃. Most of the iron in Lunar regolith appear as FeO, although the simulant (JSC-1) developed for Lunar regolith is shown to contain some Fe₂O₃ (Table 1, Column 2), since it is nearly impossible for an iron bearing soil on Earth's surface to be completely free from Fe₂O₃.

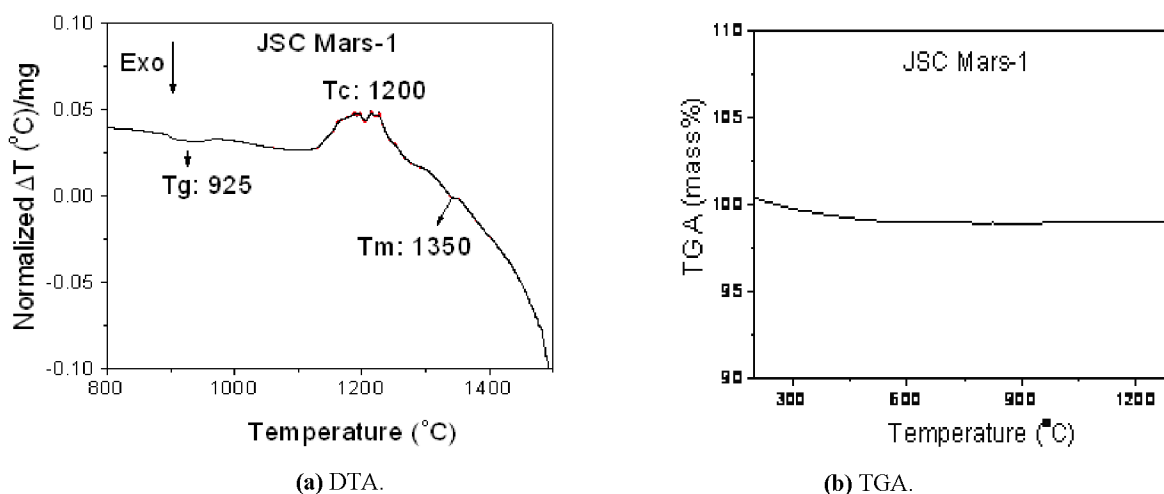


FIGURE 4. Thermal Profiles for the JSC Mars-1 Simulant.

Knowledge of the oxidation state of iron and its distribution among iron bearing minerals (e.g., olivine, pyroxene, ilmenite, magnetite, etc) in the regolith is important for mineralogical assessment, and Mössbauer spectroscopy is a powerful tool to accomplish that. The typical Mössbauer spectra measured in this study for the JSC-1 Lunar and JSC Mars-1 simulants are shown in Fig. 5. These spectra were obtained at room temperature on a spectrometer provided with a 10 mC rhodium matrix cobalt-57 source. The amount of iron found at the sample holder was 4 mg/cm². The velocity of the source was calibrated using a pure iron foil. The general appearance of the spectra for the respective simulants is similar to that reported elsewhere (Carpenter et al., 2003; Morris et al., 1998) for these simulants.

In this study three Lorentzian doublets have been used to fit each spectrum and each doublet has been assigned to the iron redox ion, Fe²⁺ and Fe³⁺, see Fig. 5. It is not exactly known at this time which iron bearing component (e.g., pyroxene, ilmenite, etc.) of the simulant is responsible for the appearance of a specific set of doublet in the Mössbauer spectra. Further analyses of the spectra including measuring the Mössbauer parameters such as quadrupole splitting and isomer shift, are continuing to identify the respective components of the simulant associated with each doublet. At this time, however, the concentration of each ion, Fe²⁺ and Fe³⁺, in the simulants was calculated from the Mössbauer spectra, and is shown in Table 2. The JSC-1 Lunar simulant contains a higher concentration, about 72%, of ferrous iron (Table 2), where as, the iron redox species in the JSC Mars-1 simulant is predominantly ferric, about 73%. These results are consistent with the average composition of the simulants given in columns 2 (McKay et al., 1994) and 4 (Allen et al., 1998) of Table 1.

TABLE 2. Fraction of Iron Calculated from the Mössbauer Spectra at 293K for JSC-1 Lunar and JSC Mars-1 Simulants.

Component	JSC-1 Lunar	JSC Mars-1
	%	%
Fe ²⁺ /(Fe ²⁺ + Fe ³⁺)	72	27
Fe ³⁺ /(Fe ²⁺ + Fe ³⁺)	28	73

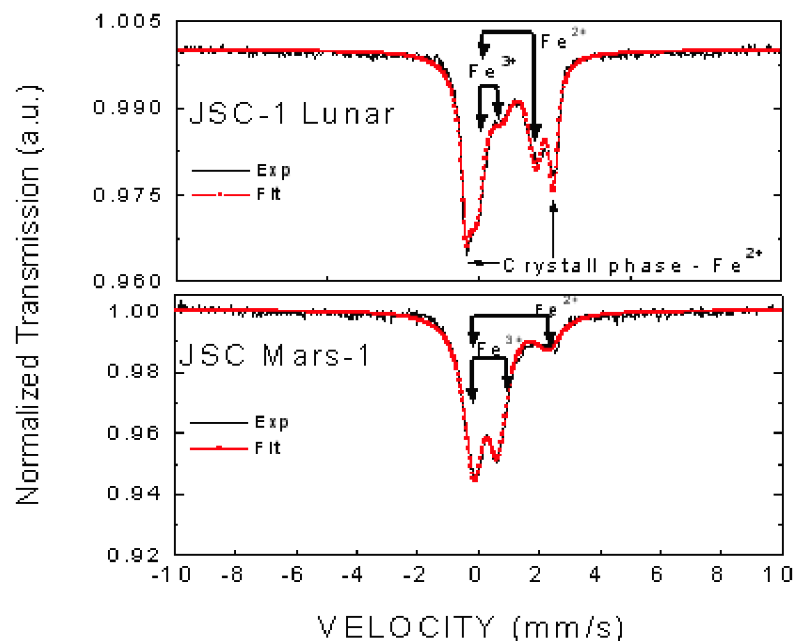


FIGURE 5. Mössbauer Spectra for the JSC-1 Lunar and JSC Mars-1 Simulants.

Critical Cooling Rate for Glass Formation (R_C)

The critical cooling rate for glass formation (R_C) is defined as the slowest rate a melt can be cooled to solid without crystallizing, i.e. the solidified melt becomes a glass. This implies that the melts with increasing values of R_C have decreasing ability to form glass on cooling. Since, preparing glass preforms and other glass-derived products from the melts of JSC-1 Lunar and Martian simulants is one of the primary goals of this program; measuring R_C for these melts is an important task.

A new experimental method that uses DTA and developed by ourselves (Ray et al., 2005) was used to measure R_C for these melts. This method which utilizes crystallization peaks obtained by heating solidified melts produced at different cooling rates (R), is rapid and easy to conduct. The detailed experimental procedure for measuring R_C by this DTA method is given in Ray et al., 2005. In brief, it requires melting the mixture of raw materials (simulant in this case) in DTA at a temperature about 150 to 200 °C higher than its melting temperature, cooling the melt at a rate R to room temperature, and finally re-heating the solidified melt at a different rate, Φ , until crystallization is complete. This experiment is repeated several times by changing R while keeping Φ constant; 15 °C/min in the present experiments. Examples of thermal profiles for a few such heating-cooling cycles for the melts of Lunar simulant are shown in Fig. 6.

The area of an exothermic DTA peak obtained on re-heating a solidified melt is proportional to the amount of heat evolved during crystallization and, hence, to the amount of residual glass present in the sample after cooling the melt, which, in turn, depends upon R , the prior cooling rate of the melt. Higher is the value of R , larger is the volume fraction of glass present in the solidified melt. A melt which partially crystallizes on cooling (for a slow R , for example) will contain a smaller fraction of glass, and its subsequent rate-heating DTA peak area will be smaller compared to that for a sample prepared by cooling the melt at a faster R . This is clearly shown in Fig. 6 for the JSC-1 Lunar simulant, where the rate-heating DTA peak area for the sample increases with increasing R that was used to quench the melt.

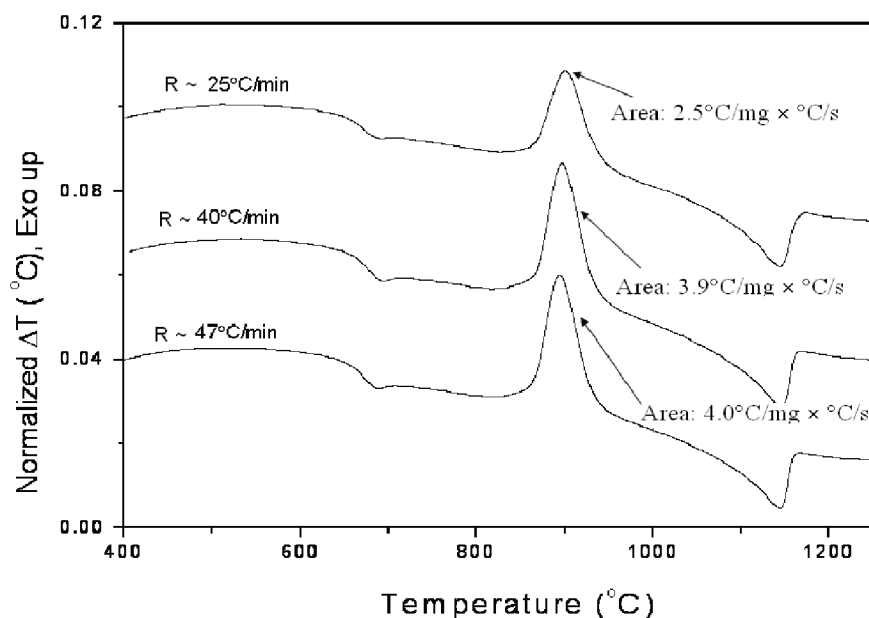


FIGURE 6. DTA Curves at a Heating Rate of 15°C/min for the Melt of JSC-1 Lunar Simulant After Quenching the Melt at Rates (R) Shown.

The DTA peak area for the samples as a function of their prior cooling rate, R , of preparation is shown in Fig. 7 for the JSC-1 Lunar simulant. Fig. 7 shows that the peak area increases initially with increasing R , which is expected per discussion above. However, it attains a plateau as the prior cooling rate of the melt exceeds a certain critical value. The area of the rate-heating DTA peak for the sample which was prepared by cooling its melt at an $R = R_C$, would be the highest, since this sample should not contain any crystal and should be totally glassy. For all samples which were prepared at $R \geq R_C$, the DTA peak area would, therefore, remain the same, Fig. 7. The value of R for which the subsequent rate-heating DTA peak area attains a maximum value is the critical cooling rate for glass formation R_C . From the analysis of Fig. 7, the value of R_C for the melt of JSC-1 Lunar simulant is determined to be about 42 ± 2 °C/min.

The melting temperature for the JSC Mars-1 simulant is much higher than that of the JSC-1 Lunar simulant, and this Martian simulant could not be completely melted even at 1500 °C (Fig. 4a), which is the maximum temperature limit of the DTA apparatus used. This means that the melt of JSC Mars-1 simulant could not be made completely crystal-free when heated at 1500 °C, and, therefore, could never be obtained as fully glass when cooled from this temperature. A furnace with a temperature capability of > 1500 °C is required for completely melting this simulant. For this reason, the value of R_C for the Martian simulant could not be determined at this time using procedures described above for the Lunar simulant. Further work on designing a suitable experimental method for measuring R_C for the JSC Mars-1 simulant is continuing.

A small amount of JSC Mars-1 simulant was melted at 1550 °C for 3 h in a glass melting furnace and the melt when cooled in ambient atmosphere appears to form glass as indicated by x-ray diffraction (XRD) analysis of the as-quenched melt (Ray et al., 2006). The glass samples shown in Fig. 8 for the Martian simulant were prepared using this procedure. At this time, it is believed that this Martian simulant needs to be heated at temperatures somewhat above 1550 °C to achieve complete melting, but once melted a moderate cooling rate between 50 and 60 °C/min may be adequate to transform the melt to glass.

A melt when cast in ambient atmosphere to produce a slab of about 1 cm thick experiences, generally, a cooling rate between 70 and 80 °C/min. If the as-cast slab turns out to be a crystal-free glass, the melt can be described as a reasonably good glass forming melt. For example, a melt of lithium disilicate ($\text{Li}_2\text{O} \cdot 2\text{SiO}_2$) composition which is commercially used to prepare glass-ceramic products, has an R_C of about 60 °C/min (Ray, Huang, and Day, 1987; Ota and Soga, 1983). By this standard, the melts of both JSC-1 Lunar and JSC Mars-1 simulants can be described as reasonably good glass forming melts. However, comparing the values of R_C of these melts, the JSC-1 Lunar simulant appears to be a better glass forming system than the JSC Mars-1 simulant.

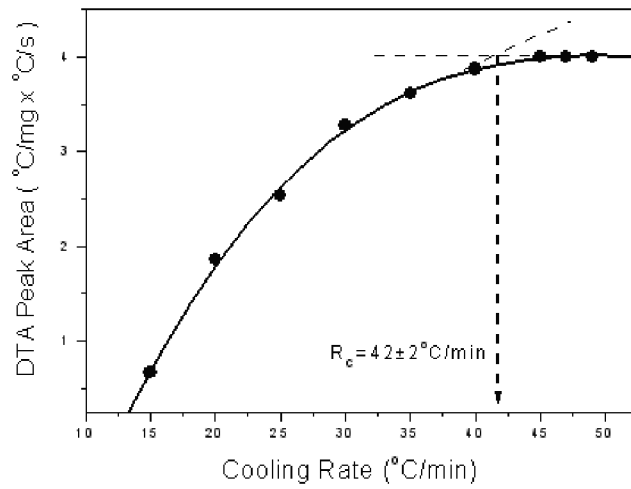


FIGURE 7. DTA Peak Area as a Function of Prior Cooling Rate for the Melts of JCS-1 Lunar Simulant.

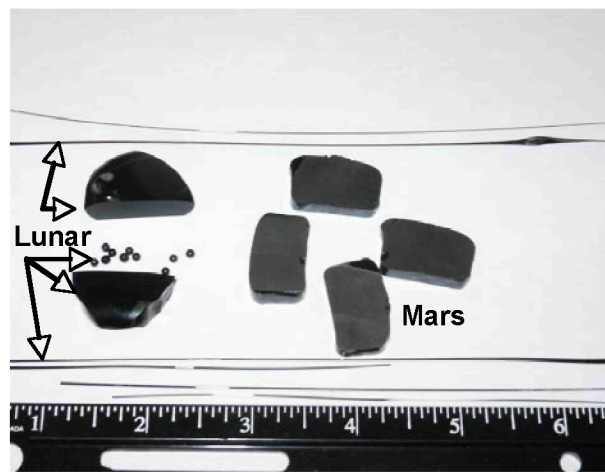


FIGURE 8. Glasses Prepared from the JSC-1 Lunar and JSC Mars-1 Simulants. Fibers and 3 mm Spheres were also Produced using JSC-1 Lunar Simulant.



FIGURE 9. Glass Fibers Prepared from JSC-1 Lunar Simulant. Typical Diameters of Individual Fibers Vary Between 15 to 30 μm .

SUMMARY

Various results that characterize the JSC-1 Lunar and JSC Mars-1 simulants have been obtained using DTA, TGA, XRD, SEM, Mössbauer spectroscopy, and chemical analysis by ICP-AES. These results, which can be used as a broad data base for these simulants will be useful for future investigations. Results from differential thermal analysis and critical cooling rate measurements show that the melts of both simulants can be transformed to glass on cooling with no major difficulty, although the glass forming ability of the Lunar simulant appears to be a little higher than that of the Martian simulant. Typical examples of a few glass preforms as prepared in an initial effort from the JSC-1 Lunar and JSC Mars-1 simulants are shown in Fig. 8. Qualitative assessment suggests that both melts have a viscosity-temperature characteristic that is suitable for pulling continuous glass fibers. An example is shown in Fig. 9, where a strand of glass fibers prepared from the melts of JSC-1 Lunar simulant has been displayed. The strand of glass fibers in Fig. 9 is a portion of a much longer (about 4 m) bundle of fibers that were prepared to demonstrate the ability to pull continuous glass fibers from the melt of JSC-1 Lunar simulant. The use of glass fibers to prepare glass fiber reinforced various composite materials for structural application is well known.

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